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IS 10581 (1983): Methods of test for the determination of ionic impurities in electrical insulating materials by extraction with liquids [ETD 2: Solid Electrical Insulating Materials and Insulation Systems]



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*Indian Standard*

METHODS OF  
TEST FOR THE DETERMINATION OF  
IONIC IMPURITIES IN ELECTRICAL  
INSULATING MATERIALS BY  
EXTRACTION WITH LIQUIDS

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**INDIAN STANDARDS INSTITUTION**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
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# Indian Standard

## METHODS OF TEST FOR THE DETERMINATION OF IONIC IMPURITIES IN ELECTRICAL INSULATING MATERIALS BY EXTRACTION WITH LIQUIDS

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*Indian Standard*

**METHODS OF  
TEST FOR THE DETERMINATION OF  
IONIC IMPURITIES IN ELECTRICAL  
INSULATING MATERIALS BY  
EXTRACTION WITH LIQUIDS**

**0. FOREWORD**

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 28 April 1983, after the draft finalized by the Solid Electrical Insulating Materials Sectional Committee had been approved by the Electrotechnical Division Council.

**0.2** The determination of ionic impurities in electrical insulating materials is of particular significance for the electrical insulating materials which are immersed in coolants/impregnants in the field application. The presence of ionic impurities which may be present as ionizable acids, bases, salts or a combination of these is confirmed by increase in volume conductivity of the liquid extract of insulating materials.

The impurities are chemical residues arising from the manufacturing process, and which have been incompletely removed. The presence of excessive amounts of electrolytic impurities is undesirable, as they tend to lower insulation resistance and have corrosion-producing tendencies under conditions of applied potential.

**0.3** In the preparation of this standard assistance has been derived from IEC Pub 589 ( 1977 ) 'Methods of test for the determination of ionic impurities in electrical insulating materials by extraction with liquids', issued by the International Electrotechnical Commission.

**0.4** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960\*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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\*Rules for rounding off numerical values ( revised ).

## 1. SCOPE

**1.1** This standard covers methods of test for determining the presence of ionizable soluble organic and/or inorganic materials in electrical insulating materials.

**1.2** The presence of ionic impurities is confirmed by the increase in volume conductivity of the liquid extract.

## 2. TERMINOLOGY

**2.0** For the purpose of this standard, the following definitions shall apply.

**2.1** Volume conductivity of a liquid extract ( $\gamma_{ex}$ ) is the difference between the volume conductivity of the extract and volume conductivity of the control sample ( blank sample ).

NOTE — The SI unit of the volume conductivity is  $S.m^{-1}$ . In practice the unit  $S.cm^{-1}$  is often used.

## 3. APPARATUS

**3.1** The following apparatus are required:

**3.1.1 Conductivity Bridge** — Or any other measuring device capable of measuring resistance up to 1 000 000 ohms with an accuracy of 5 percent at frequencies between 50 Hz and 3 000 Hz.

**3.1.2 Conductivity Cell** — A dip-type cell with platinum electrodes securely mounted and adequately protected so that their relative positions will not be affected by handling or moderate jarring, shall be used. The area of each electrode shall be not less than 2 cm<sup>2</sup>. The cell shall be so constructed that electrodes will be completely immersed on dipping the cell into the liquid medium. The cell constant shall be 0.1 cm<sup>-1</sup>. The electrodes shall be platinized as per method given in Appendix A, if the measurements are made at low frequency ( 50 Hz ). At a frequency of 1 kHz this precaution is unnecessary. The conductivity cell shall be calibrated as given in Appendix A.

**3.1.3 Motor and Stirrer** — A motor capable of operating at 5 000 rpm or higher speed, fitted with a stirrer, which shall be made of brass and shall be chromium plated, to render it acid and alkali resistant.

**3.1.4 Constant-Temperature Bath** — A water bath maintained at  $27 \pm 0.5^{\circ}C$ .

**3.1.5 Beaker** — 125-ml beakers made of acid and alkali-resistant glass, tall-form or of such dimensions that when the dip cell is immersed in 100 ml of liquid contained therein, the electrodes are fully covered.



**3.1.6 Flasks** — Wide-mouth, 250 ml Erlenmeyer flasks, made of acid and alkali resistant glass.

**3.1.7 Suction Filtration Apparatus**

**3.1.8 Perforated Disk** — A perforated porcelain disk, 50 mm in diameter, with its edge bevelled at an angle of 60°. It shall have approximately 90 perforations, and the diameter of the perforations shall be approximately 1 mm.

**3.1.9 Funnel** — An acid and alkali-resistant glass funnel having a top diameter of 100 mm and made with an exact 60° angle.

**3.1.10 Thermometers** — One thermometer having a range from — 10 to 110°C and graduated in 1°C intervals ( for extract solution ), and one thermometer having a range from — 5 to + 5°C and graduated in 0.1°C intervals ( for constant-temperature bath ).

**3.1.11 Hot Plate** — electric.

#### 4. PREPARATION OF THE AQUEOUS EXTRACT

**4.1** Unless specified otherwise, the aqueous extract shall be prepared as follows:

**4.1.1 Test Water** — The quality of the test water has a considerable influence on the result of the test. The volume conductivity of the test water preparing the aqueous extracts shall be  $\leq 2 \times 10^{-4} \text{ S.m}^{-1}$ . The pH value of the test water shall be between 6.8 and 7.2. The test water can be made by means of an ion-exchanger or by using double distilled water of the recommended pH value.

NOTE — Attention is drawn to the effect of carbon dioxide, the pH value of newly prepared water will change to between 5.7 and 5.9 after the water has been exposed to air for a short time. If the change due to carbon dioxide has produced a pH of not less than 6.4, the water can be restored to pH 7 by blowing pure nitrogen through it for a short time. The pH will remain within the required limit for a long time, if the water is stored in polyethylene flasks from which carbon dioxide is excluded.

**4.2 Procedure** — Take a test specimen weighing approximately 20 g, cut into pieces of suitable dimensions. Weigh  $5 \pm 0.1 \text{ g}$  of these small pieces each into three flasks fitted with reflux condensers and add 100 ml of test water. A fourth flask and condenser shall be used for the blank control sample.

The water in the four flasks shall be kept boiling gently for  $60 \pm 5$  minutes and then cooled in the flask to room temperature. Precautions shall be taken to avoid admission of carbon dioxide.

## 5. PREPARATION OF ORGANIC EXTRACT

**5.1 Test Liquid** — If it is necessary to use organic liquids for the determination of ionic impurities in electrical insulating materials, trichloroethylene is suitable, and shall be used unless specified otherwise. The volume conductivity of trichloroethylene for preparing the liquid extracts shall be  $0.5 \times 10^{-9} \text{ S.m}^{-1}$ . For special cases,  $1 \cdot 10^{-11} \text{ S.m}^{-1}$  may be used. The solvent is usually of laboratory reagent grade. Therefore, it shall be purified by stirring in about 1 percent by mass of Fuller's earth and filtering through a sintered glass filter. A filter having a maximum bore diameter in the range of 5  $\mu\text{m}$  to 15  $\mu\text{m}$  is suitable.

NOTE 1 — Fullers' earth becomes ineffective if allowed to absorb moisture and may be dried by heating in clean air at a temperature not exceeding 120°C.

NOTE 2 — Purified trichloroethylene is stable if kept in the dark or in a brown bottle, but its conductivity shall be checked before use for an extraction. During extraction and measurement the solvent should be shielded from strong light, especially direct sunlight, and stored in the dark.

NOTE 3 — Trichloroethylene is toxic and should be handled with appropriate care.

**5.2 Procedure** — Cut a test specimens weighing approximately 20 g into pieces of suitable dimensions and heat them gently in air for about 2 hours at a temperature of 80 to 100°C, in order to remove any appreciable amount of absorbed water. Weigh  $5 \pm 0.1$  g of these small pieces into three flasks fitted with reflux condensers, as described in 3, and add 100 ml of solvent. A fourth flask and condenser shall be used for the control sample.

The solvent in the four flasks shall be boiled gently for  $60 \pm 5$  minutes and then cooled in the flasks to room temperature. The whole process shall be carried out in the dark.

NOTE — The loss of volume through evaporation shall not exceed 10 percent, otherwise, the test shall be repeated.

## 6. MEASUREMENTS

**6.1** Before measuring the resistance of extracts, the resistance of the control sample shall be determined.

**6.1.1** The trichloroethylene extract shall be decanted from the test specimens into the conductivity cell for measuring immediately.

**6.1.2** The cell shall have been cleaned several times with purified trichloroethylene and dried before filling. After the cell has been rinsed twice with extract, the measurement of the resistance shall be carried out on each extract separately.

**6.1.3** The measurement shall be carried out 1 minute after application of the dc voltage at  $27 \pm 1^\circ\text{C}$ .

NOTE — It is essential to ensure that during the drawal, storage and manipulation of samples, specimens and test portions intended for the conductivity test of organic extract, these are not contaminated either by the atmosphere, particularly the atmosphere of the chemical laboratory, or during handling with bare hands.

## 7. CALCULATION

**7.1** The volume conductivity of the liquid extract shall be calculated by:

$$\gamma_{\text{ex}} = \frac{1}{Q_{\text{ex}}} = k \left( \frac{1}{R_s} - \frac{1}{R_B} \right)$$

where

$\gamma_{\text{ex}}$  = volume conductivity of the liquid extract in  $\text{S.m}^{-1}$  ( $\text{S.cm}^{-1}$ ),

$Q_{\text{ex}}$  = volume resistivity of the liquid extract in  $\Omega.\text{m}$  ( $\Omega.\text{cm}$ ),

$k$  = cell constant in  $\text{m}^{-1}$  ( $\text{cm}^{-1}$ ),

$R_s$  = resistance of the extract solution in  $\Omega$ , and

$R_B$  = resistance of the control sample in  $\Omega$ .

## 8. TEST REPORT

**8.1** The test report shall include the following information:

- Designation of the electrical insulating material,
- Designation of the liquid,
- All test results, and
- Date of test.

## A P P E N D I X A

( Clause 3.1.2 )

### PREPARATION AND CALIBRATION OF CONDUCTIVITY CELL

**A-1.** If the electrodes are unplatinized, clean a new cell with warm chromic acid solution wash thoroughly with reagent water, and rinse with alcohol and ether. If the electrodes are already platinized, omit the chromic acid wash.

**A-2.** To platinize the electrodes immerse the cell in solution of 3.0 g of chloroplatinic acid and 0.010 g of lead acetate in 100 ml of reagent water. Electrolyze, using a current density of 30 mA/cm<sup>2</sup> for 8 minutes reversing the current every 2 minutes, wash the electrodes thoroughly with reagent water. To test for completeness of removal of electrolyte, immerse the cell in 50 ml of reagent water and measure the resistance initially and at the end of 10 minutes. If a decrease in resistance occurs, repeat the washing. Keep the cell immersed in reagent water when not in use.

**A-3.** To determine the cell constant place a breaker containing 0.01 N KCl, solution in the constant temperature bath maintained at  $27 \pm 0.5^\circ\text{C}$ . After thermal equilibrium is established, measure the resistance of this solution. The cell constant,  $k$  shall be calculated as follows:

$$k = C \times R$$

where

$R$  = resistance measured, and

$C$  = conductivity of the potassium chloride solution. The value for  $C$ , at  $27^\circ\text{C}$  is  $1.41 \times 10^{-3} \text{ S.cm}^{-1}$ .